

Priority number(s): JP20030039352
20030218;
JP20020042586
20020220

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Abstract of JP 2003313027 (A)

PROBLEM TO BE SOLVED: To provide a method of producing a boehmite sol by which a recording medium capable of performing recording of high quality can be produced on information recording by an ink jet process, to provide a boehmite sol, to provide a method of producing a recording medium using the same, and to provide a recording medium. ; **SOLUTION:** A boehmite sol used for the production of a recording medium for an ink jet is produced. In this case, amorphous alumina hydrate slurry prepared by neutralization reaction between a basic aluminum chloride aqueous solution and a sodium aluminate aqueous solution, preferably under the condition of ≤ 40 [deg.]C is subjected to hydrothermal treatment in an alkali region of pH ≥ 10 at ≥ 120 [deg.]C to form boehmite slurry. Thereafter, the slurry is deflocculated to form a boehmite sol. The viscosity of the boehmite sol in the case the sol concentration is controlled to 20 wt.% expressed in terms of Al_2O_3 is ≤ 100 cP. Also the mean fine pore size of pores formed in boehmite fine powder obtained by drying the boehmite sol is ≥ 15 nm. The volume of the pores is ≥ 0.5 cc/g. ; **COPYRIGHT:** (C)2004,JPO

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Family list

1 application(s) for: JP2003313027 (A)

**1 METHOD OF PRODUCING BOEHMITE SOL,
BOEHMITE SOL, METHOD OF PRODUCING
RECORDING MEDIUM AND RECORDING
MEDIUM**

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B41J2/01;
B41M5/00;
B41M5/50;
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Publication JP2003313027 (A) - 2003-11-06
info: JP4368118 (B2) - 2009-11-18

Priority
Date: 2002-
02-20

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Machine Translation of "G"

JP,2003-313027,A

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[Claim(s)]

[Claim 1]Adjust a hydrated alumina slurry with a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution, and this hydrated alumina slurry in pH ten or more alkali fields. And after performing water heat treatment at temperature of not less than 120 ** and obtaining a boehmite slurry, carrying out amalgam decomposition of the boehmite slurry concerned -- boehmite -- in obtaining sol, When adjusting said hydrated alumina slurry with a neutralization reaction with said basic aluminum chloride solution and said sodium aluminate solution, boehmite adding said basic aluminum chloride solution to said sodium aluminate solution -- a manufacturing method of sol.

[Claim 2]boehmite setting it as conditions which become higher than pH of said hydrated alumina slurry before pH of a boehmite slurry after performing said water heat treatment performs the water heat treatment concerned in Claim 1 -- a manufacturing method of sol.

[Claim 3]A hydrated alumina slurry prepared by neutralization reaction with basic aluminum chloride solution and sodium aluminate solution in pH ten or more alkali fields. And after performing water heat treatment at temperature of not less than 120 ** and obtaining a boehmite slurry, carrying out amalgam

decomposition of the boehmite slurry concerned -- boehmite -- boehmite setting it as conditions which become higher than pH of said hydrated alumina slurry before pH of a boehmite slurry after performing said water heat treatment performs the water heat treatment concerned in obtaining sol -- a manufacturing method of sol.

[Claim 4]After adjusting an amorphous hydrated alumina slurry with a neutralization reaction with said basic aluminum chloride solution and said sodium aluminate solution in either of the Claims 1-3, boehmite performing said water heat treatment to the amorphous hydrated alumina slurry concerned -- a manufacturing method of sol.

[Claim 5]boehmite performing said neutralization reaction at temperature of 40 °C or less in either of the Claims 1-4 -- a manufacturing method of sol.

[Claim 6]boehmite, wherein a Cl/aluminum mole ratio of a basic aluminum chloride is in ranges from 0.45 to 1.5 in either of the Claims 1-5 -- a manufacturing method of sol.

[Claim 7]boehmite manufacturing by a method of specifying to either of the Claims 1-6 -- sol.

[Claim 8]boehmite, wherein viscosity when sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration in Claim 7 is 100 or less cP -- sol.

[Claim 9]boehmite, wherein viscosity when said sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration in Claim 8 is 50 or less cP -- sol.

[Claim 10]boehmite characterized by said sol concentration being 18 % of the weight or more by aluminum₂O₃ conversion concentration in Claim 8 or 9 -- sol.

[Claim 11]in either of the Claims 7-10 -- boehmite -- boehmite in which an average pore size of fine pores formed in boehmite impalpable powder produced by drying sol is characterized by pore volume being 0.5 or more cc/g at not less than 15 nm -- sol.

[Claim 12]boehmite specified to either of the Claims 7-11 -- a manufacturing method of a recording medium having made it

dry and forming a porous layer by boehmite hydrate particles in said base material surface after applying on a substrate coating liquid which mixed and prepared a binder to sol.

[Claim 13]boehmite specified to either of the Claims 7-10 -- a recording medium which makes it dry, forms a porous layer by boehmite hydrate particles in said base material surface after applying on a substrate coating liquid which mixed and prepared a binder to sol, and is characterized by things.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]boehmite suitable for this invention manufacturing the recording medium used for Information Storage Division by the ink jet method etc. -- sol and its manufacturing method, and this boehmite -- it is related with the recording medium using sol, and its manufacturing method.

[0002]

[Description of the Prior Art]The recording medium which formed the ink absorbing layer containing hydrated alumina particles on the substrate as a recording medium used for Information Storage Division by the ink jet method is known. This ink absorbing layer is a dried porous layer, after applying on a substrate the coating liquid containing alumina sol. In such an ink absorbing layer, the various characteristics are required from an ink absorbing layer -- if the pore diameter or pore volume of hydrated alumina particles are not large enough, ink absorption time is long, and overflow, a blot, etc. of ink occur.

[0003]Here, as a manufacturing method of alumina sol, after hydrolyzing aluminum isopropoxide conventionally, the method of adding and carrying out amalgam decomposition of the acid, the method of riping and solating the alumina slurry which hydrolyzed and obtained aluminum DODEKISHIDO, etc. are known.

[0004]

[Problem(s) to be Solved by the Invention]However, after applying the coating liquid using the conventional alumina sol on a substrate, the pore radius and pore volume of hydrated

alumina particles are small, and the ink absorbency of the recording medium which made it dry and formed the porous layer by hydrated alumina particles on the substrate is insufficient.

[0005] If coating liquid is prepared using the alumina sol obtained with the conventional manufacturing method when manufacturing a recording medium, since viscosity is high, it is difficult for a base material surface to carry out a coat homogeneously. Therefore, in order to have to make sol concentration of coating liquid low conventionally, there is also a problem of being unable to manufacture a recording medium efficiently.

[0006] the boehmite which fitted manufacture of the recording medium which can perform high record of grace when SUBJECT of this invention performed Information Storage Division by the ink jet method in view of the above problem -- the manufacturing method of sol, and boehmite -- it is in providing sol, the manufacturing method of the recording medium using it, and a recording medium.

[0007]

[Means for Solving the Problem] a result in which an invention-in-this-application person inquired repeatedly in order to solve an aforementioned problem -- the following boehmites -- when having manufactured a recording medium using sol and Information Storage Division by the ink jet method was performed, high record of grace could be performed and knowledge that a recording medium could be manufactured suitably was acquired.

[0008] namely, boehmite of this invention -- in a manufacturing method of sol. Adjust a hydrated alumina slurry with a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution, and this hydrated alumina slurry in pH ten or more alkali fields. And after performing water heat treatment at temperature of not less than 120 ** and obtaining a boehmite slurry, carrying out amalgam decomposition of the boehmite slurry concerned -- boehmite -- in obtaining sol, When adjusting said hydrated alumina slurry

with a neutralization reaction with said basic aluminum chloride solution and said sodium aluminate solution, said basic aluminum chloride solution is added to said sodium aluminate solution.

[0009]boehmite -- although sol is marketed also from the former, the conventional commercial item has sol concentration as low as 5 to 10 % of the weight, in order to stop viscosity, and if such sol is condensed for high-concentration-izing, viscosity will rise, and there is a problem of gelling by temporality. therefore, the conventional boehmite -- the time of manufacturing a recording medium using sol -- a base material surface -- boehmite -- it is difficult homogeneity and to carry out the coat of the sol efficiently. However, in this invention, after preparing a hydrated alumina slurry, to this hydrated alumina slurry in pH ten or more alkali fields. and -- obtaining a boehmite slurry which performed water heat treatment at a temperature as high as not less than 120 **, and made boehmite generate quickly, and carrying out amalgam decomposition of this boehmite slurry -- boehmite -- sol is obtained. for this reason -- a time of, for example, making sol concentration into 20 % of the weight by $\text{aluminum}_2\text{O}_3$ conversion concentration -- viscosity -- 100 or less cP and boehmite whose viscosity of 50 or less cP is still lower -- sol can be obtained. And when a hydrated alumina slurry is adjusted with a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution in this invention, In order to add basic aluminum chloride solution to sodium aluminate solution, as compared with a case where sodium aluminate solution is added to basic aluminum chloride solution, hydrated alumina slurry concentration obtained by neutralization can be raised -- high-concentration boehmite -- it is suitable for manufacturing sol. so, boehmite concerning this invention -- even when manufacturing a recording medium, and using sol and coating liquid is prepared [a binder etc. are added and], sol concentration in coating liquid can be kept high. If such coating liquid is used, such a thick coat is obtained, and since

the volumetric shrinkage in the case of desiccation is small, there is an advantage, like a crack cannot go into a film easily that sol concentration is high. Since concentration can be raised by filtration enrichment, there is also an advantage of not needing an expensive concentration device called a vacuum concentration device etc.

[0010]Here, basic aluminum chloride solution and sodium aluminate solution are used for preparing a hydrated alumina slurry because a homogeneous hydrated alumina slurry can be obtained by those neutralization reactions. For example, when aluminum sulfate is used as an aluminum raw material, sulfate ion cannot remain in hydrated alumina, and a hydrated alumina slurry suitable for boehmite-ization cannot be obtained. If NaOH is used instead of sodium aluminate as an alkali raw material, a part of bayerite (aluminum (OH)₃) will generate, and a homogeneous hydrated alumina slurry will not be obtained.

[0011]moreover -- if carrying out water heat treatment of the hydrated alumina above 120 °C performs hydrothermal reaction by such high temperature conditions, a boehmite crystal will grow uniformly and promptly -- boehmite of homogeneous and stable hypoviscosity -- it is because sol can be obtained. when hydrothermal temperature is 120 °C or less, in order that [on the other hand,] bayerite of coarse grain may generate selectively -- homogeneous boehmite -- sol cannot be prepared. homogeneous and stable boehmite to which boehmite particles were equal when boehmite-izing a hydrated alumina slurry at temperature of not less than 120 °C for a short time -- sol is obtained.

[0012]carrying out water heat treatment of the hydrated alumina slurry or more by pH ten -- high concentration -- boehmite of hypoviscosity -- boehmite which can obtain sol and can prepare boehmite powder with big pore diameter and pore volume by desiccation -- it is because sol can be obtained. on the other hand, when pH is lower than ten, it mentions later as the comparative example 1 -- as -- boehmite of hypoviscosity -- boehmite powder which has sol, sufficient pore diameter,

and pore volume cannot be obtained.

[0013] In this invention, it is preferred to perform said water heat treatment on conditions which become higher than pH of said hydrated alumina slurry before pH of a boehmite slurry after performing said water heat treatment performs the water heat treatment concerned. For example, if water heat treatment is performed on condition of this invention using superfluous sodium aluminate from a neutralization equivalent with a basic aluminum chloride, pH of a slurry after hydrothermal reaction will rise and generation in a short time of boehmite and uniform particle growth will happen. Although this cause is not yet clear, it is thought that boehmite particles grow easily uniformly with a rise of pH under water heat treatment.

[0014] boehmite of this invention -- in a manufacturing method of sol. A hydrated alumina slurry prepared by neutralization reaction with basic aluminum chloride solution and sodium aluminate solution in pH ten or more alkali fields. and -- carrying out amalgam decomposition of the boehmite slurry concerned, after performing water heat treatment at temperature of not less than 120 °C and obtaining a boehmite slurry -- boehmite, while obtaining sol, It is set as conditions which become higher than pH of said hydrated alumina slurry before pH of a boehmite slurry after performing said water heat treatment performs the water heat treatment concerned.

[0015] In this invention, if a hydrated alumina slurry is adjusted with a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution, a hydrated alumina slurry can be obtained, but if such a hydrated alumina slurry is neglected, a part will crystallize. If water heat treatment is performed on conditions which become higher than pH of a hydrated alumina slurry before pH of a boehmite slurry after performing water heat treatment performs water heat treatment even if it is such a hydrated alumina slurry, generation in a short time of boehmite and uniform particle growth will happen. For example, if water heat treatment is performed on condition of this invention using superfluous

sodium aluminate from a neutralization equivalent with a basic aluminum chloride, pH of a slurry after hydrothermal reaction will rise and generation in a short time of boehmite and uniform particle growth will happen. Although this cause is not yet clear, it is thought that boehmite particles grow easily uniformly with a rise of pH under water heat treatment.

[0016] In this invention, after preparing a hydrated alumina slurry, to this hydrated alumina slurry in pH ten or more alkali fields. and -- obtaining a boehmite slurry which performed water heat treatment at a temperature as high as not less than 120 **, and made boehmite generate quickly, and carrying out amalgam decomposition of this boehmite slurry -- boehmite, in order to obtain sol, a time of making for example, sol concentration into 20 % of the weight by aluminum₂O₃ conversion concentration -- viscosity -- 100 or less cP and boehmite whose viscosity of 50 or less cP is still lower -- sol can be obtained. so, boehmite concerning this invention -- even when manufacturing a recording medium, and using sol and coating liquid is prepared [a binder etc. are added and], sol concentration in coating liquid can be kept high. If such coating liquid is used, such a thick coat is obtained, and since the volumetric shrinkage in the case of desiccation is small, there is an advantage, like a crack cannot go into a film easily that sol concentration is high. Since concentration can be raised by filtration enrichment, there is also an advantage of not needing an expensive concentration device called a vacuum concentration device etc.

[0017] It is because using basic aluminum chloride solution and sodium aluminate solution for preparing a hydrated alumina slurry here can obtain a homogeneous hydrated alumina slurry by those neutralization reactions as described above. carrying out water heat treatment of the amorphous hydrated alumina above 120 ** -- said -- as carried out, a boehmite crystal grows uniformly and promptly -- boehmite of homogeneous and stable hypoviscosity -- it is because sol can be obtained. homogeneous and stable boehmite to which boehmite particles were equal

since a hydrated alumina slurry was boehmite-ized at temperature of not less than 120 ** for a short time -- sol is obtained. furthermore -- since water heat treatment of the amorphous hydrated alumina slurry is carried out or more by pH ten -- high concentration -- boehmite of hypoviscosity -- sol can be obtained -- and desiccation -- boehmite which can prepare boehmite powder with big pore diameter and pore volume -- sol can be obtained.

[0018] In this invention, it is preferred to adjust an amorphous hydrated alumina slurry and to perform said water heat treatment to the amorphous hydrated alumina slurry concerned by neutralization reaction with said basic aluminum chloride solution and said sodium aluminate solution. a direction which an amorphous hydrated alumina slurry could be adjusted when basic aluminum chloride solution and sodium aluminate solution were counteracted, but performed water heat treatment to an amorphous hydrated alumina slurry -- boehmite of high concentration and hypoviscosity -- sol can be obtained certainly.

[0019] boehmite concerning this invention, in order to manufacture a recording medium corresponding to an ink jet using sol, for example, boehmite -- after applying on a substrate coating liquid which mixed and prepared a binder to sol, it is made to dry and a porous layer by boehmite hydrate particles is formed in said base material surface as an ink absorbing layer.

[0020] It is preferred to perform said neutralization reaction below 40 ** by this invention. If it counteracts at such a low temperature, an amorphous hydrated alumina slurry will be obtained. on the other hand -- when reaction temperature is not less than 40 **, in order that a part of bayerite of coarse grain and low crystalline boehmite may generate, even if it carries out water heat treatment of this hydrated alumina slurry -- homogeneous and stable boehmite -- it is in a tendency for sol not to be obtained.

[0021] In this invention, it is preferred that a Cl/aluminum

mole ratio of a basic aluminum chloride is in ranges from 0.45 to 1.5. In basicity of an aluminium chloride, since it will become quiet compared with a case where a reaction with sodium aluminate is normal salt (AlCl_3) if basicity is given, homogeneous amorphous hydrated alumina is obtained. Quantity of a salt (NaCl) generated compared with a neutralization reaction of normal salt at the time of a neutralization reaction can be lessened substantially. On the other hand, since it becomes easy to generate bayerite when a Cl /aluminum mole ratio is high (i.e., when basicity is low), it is not desirable. [0022]viscosity when sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration according to the manufacturing method concerning this invention -- boehmite of 100 or less cP -- sol can be obtained. boehmite -- the time of manufacturing a recording medium using sol -- boehmite -- in order to mix a binder to sol, to prepare coating liquid and to apply it on a substrate, it is preferred that viscosity when said sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration in this invention is 50 or less cP. It is preferred that said sol concentration is 18 % of the weight or more by aluminum₂O₃ conversion concentration.

[0023]moreover -- this invention -- boehmite -- boehmite whose pore volume an average pore size of fine pores formed in boehmite impalpable powder produced by drying sol is 0.5 or more cc/g in not less than 15 nm -- it is preferred to manufacture sol. such boehmite -- not less than 15 nm and pore volume are the ranges of 0.5 - 1.0 cc/g, and pore diameters can use boehmite powder produced by drying sol conveniently for a recording medium for inkjet printing paper. Pore volume distribution of this boehmite powder is dramatically sharp. Specifically, capacity of fine pores which enter within the limits of ≤ 10 nm of pore diameters in a peak position of pore volume distribution occupies not less than 65% of the whole pore volume. This is considered to originate in particle diameter of boehmite particles having gathered.

[0024]in this invention -- boehmite -- as for pH of sol, the 3.5-4.5th place is preferred. When pH is high, viscosity rises. On the other hand, when pH is low, a part of aluminum comes to dissolve and it is not desirable. Although acid in particular used as a dispersing agent which adjusts pH is not limited, carboxylic acid, such as inorganic acid, such as nitric acid and chloride, or acetic acid, can be used conveniently.

[0025]

[Example] Hereafter, this invention is explained still in detail. Measurement of the various physical properties in connection with this invention was performed by the following methods.

[0026] Identification of a crystal form: It measured using X-ray diffractometer (made by RINT2000 Rigaku Corp.) about the hydrated alumina before and behind water heat treatment.

[0027] the pore volume of a dried sample, an average pore size, and the boehmite dried at pore-volume-distribution: 60 ** -- under evacuation, at 150 **, after [2 hours] carrying out degassing treatment, it measured about sol using the nitrogen absorption desorption device (ASAP2000 made from Micromeritics). Pore volume is a value 1.7-300 nm in diameter, and pore distribution was searched for by the BJH method.

[0028] Sol viscosity: After adjusting sol temperature to 20 **, it measured using Brookfield viscometer (made in Tokyo Keiki factory) NO.1 rotor.

[0029] sol concentration: -- boehmite -- after adding and carrying out the heating and dissolving of the acid to sol, Al content was calculated with chelatometry, and it converted into aluminum₂O₃ concentration.

[0030] transmissivity: -- the boehmite which made sol concentration 0.5 % of the weight -- the transmissivity of light with a wavelength of 530 nm was measured for sol in 10 mm of light path length's cell.

[0031] Although working example 1-9 and the comparative examples 1-3 are explained below among examination which the invention-in-this-application person performed repeatedly,

about working example 1-5 and the comparative examples 1 and 2, the condition and an evaluation result are shown in Table 1 among these examples.

[0032]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	比較例 1	比較例 2
細孔容積 cc/g	0.753	0.787	0.704	0.695	0.684	0.444	バイヤライト生成
細孔直径 nm	27.9	29.4	31.6	24.8	20.1	9.0	
Al ₂ O ₃ 濃度 %	20.2	20.1	20.2	20.2	20.1	19.1	
粘度 cP	33	30	28	36	38	330	
インク吸収性	○	○	○	○	○	×	

[0033] [Working example 1] To 5000-ml glass beakers, aluminum₂O₃ conversion concentration =23.6%, Cl/aluminum mole ratio = until pH is set to 10.6, taking the basic aluminum chloride solution (trade name: made in [Daimei Telecom Engineering Chemical industry] the alpha yne 83) of 0.501, and optimum dose of water, and stirring this solution by a homomixer, Sodium aluminate solution (aluminum₂O₃ conversion concentration = 1% and Na/aluminum mole-ratio =2.0) is gradually added under a room temperature, and an amorphous hydrated alumina slurry is obtained.

[0034] Next, hydrothermal reaction of 8 hours is performed for this slurry at 150 ** using autoclave. It was 12.0 when pH of the slurry (boehmite slurry) after water heat treatment was measured.

[0035] Next, filtration washing of the slurry after hydrothermal reaction is carried out until the electrical conductivity of filtrate becomes below 20microS/cm. thus -- adding and carrying out amalgam decomposition of optimum dose of water, after adding nitric acid of 1N so that pH may be set to four to the obtained filter cake -- boehmite -- sol is prepared.

[0036] thus, the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.2%. this boehmite -- it was 33cP when the viscosity of sol was measured. Transmissivity was 3.0% when sol concentration was made into 0.5 % of the weight. this

boehmite -- the hydrated alumina produced by drying sol is boehmite (AlOOH).

The average pore diameters were 27.9 nm, and pore volume was 0.753 cc/g.

The capacity of the fine pores which enter within the limits of ± 10 nm of the pore diameters in the peak position of pore volume distribution formed 73% of the whole pore volume.

[0037] thus, the manufactured boehmite -- for manufacturing the recording medium for ink jet recording using sol -- first -- boehmite -- 22.2g of polyvinyl alcohol (97% of degree of saponification, degree of polymerization 1700) solution is mixed 10% of the weight to 100 g of sol, and coating liquid is prepared. Next, by a bar coating machine, it applies and this coating liquid is dried in the 200-micrometer-thick high-quality paper so that the thickness after desiccation may be set to 20 micrometers, and the porous layer by boehmite hydrate particles manufactures the recording form (recording medium) formed as an ink absorbing layer.

[0038] As a result, generating of a crack etc. was not observed in a porous layer. As a result of using Epson ink jet printer PM700C for this recording form, performing test printing and checking a printing state visually, there is no blot and it was checked that ink absorbency is large.

[0039] [working example 2] -- performing the same processing as working example 1 except having made water heat treatment by autoclave into 10 hours at 150 $^{\circ}\text{C}$ -- boehmite -- sol was prepared. The aluminum₂O₃ conversion concentration of the obtained sol was 20.1%, and viscosity was 30cP. The hydrated alumina produced by drying this sol was boehmite, those average pore diameters were 29.4 nm, and pore volume was 0.787 cc/g.

[0040] As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0041] [working example 3] -- performing the same processing as working example 1 except having made water heat treatment

by autoclave into 5 hours at 170 °C -- boehmite -- sol was prepared. The aluminum₂O₃ conversion concentration of the obtained sol was 20.2%, and viscosity was 28cP. The hydrated alumina produced by drying this sol was boehmite, those average pore diameters were 31.6 nm, and pore volume was 0.704 cc/g. [0042] As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0043] [Working example 4] To 5000-ml glass beakers, aluminum₂O₃ conversion concentration = 23.0%, Cl/aluminum mole ratio = until pH is set to 10.2, taking the basic aluminum chloride solution (trade name: made in [Daimei Telecom Engineering Chemical industry] the alpha yne 75) of 0.751, and optimum dose of water, and stirring this solution by a homomixer, Sodium aluminate solution (aluminum₂O₃ conversion concentration = 1% and Na/aluminum mole-ratio = 2.0) is gradually added under a room temperature, and an amorphous hydrated alumina slurry is obtained. Next, hydrothermal reaction of 2 hours is performed for this slurry at 170 °C using autoclave. It was 11.1 when pH of the slurry after hydrothermal reaction was measured. Next, filtration, washing of the slurry after hydrothermal reaction is carried out until the electrical conductivity of filtrate becomes below 20microS/cm. thus -- adding and carrying out amalgam decomposition of optimum dose of water, after adding nitric acid of 1N so that pH may be set to four to the obtained filter cake -- boehmite -- sol is prepared.

[0044] thus, the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.2%, and viscosity was 36cP. this boehmite -- the hydrated alumina produced by drying sol was boehmite, those average pore diameters were 24.8 nm, and pore volume was 0.695 cc/g.

[0045] As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0046] [working example 5] -- performing the same processing as working example 1 except having made water heat treatment by autoclave into 10 hours at 130 ** -- boehmite -- sol was prepared. The aluminum₂O₃ conversion concentration of the obtained sol was 20.1%, and viscosity was 45cP. The hydrated alumina produced by drying this sol was boehmite, those average pore diameters were 20.1 nm, and pore volume was 0.684 cc/g. Transmissivity was 3.4% when sol concentration was made into 0.5 % of the weight.

[0047] As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0048] [Working example 6] In adjusting a hydrated alumina slurry with a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution, in working example 1, added sodium aluminate solution to basic aluminum chloride solution, but. In this gestalt, said basic aluminum chloride solution is added to sodium aluminate solution so that it may explain below.

[0049] Taking sodium aluminate solution (aluminum₂O₃ conversion concentration = 1.3% and Na/aluminum mole-ratio =2.0) to 5000-ml glass beakers, and stirring this solution with an agitator. Basic aluminum chloride solution (Cl/aluminum mole-ratio =0.501) was gradually dropped under the room temperature, and the amorphous hydrated alumina slurry of 3% of aluminum₂O₃ conversion concentration was obtained until pH was set to 10.4.

[0050] Next, hydrothermal reaction of 17 hours is performed for this slurry at 120 ** using autoclave. It was 11.7 when pH of the hydrothermal slurry was measured.

[0051] Next, filtration washing of the slurry after hydrothermal reaction was carried out until the electrical conductivity of filtrate became below 20microS/cm. thus -- adding and carrying out amalgam decomposition of optimum dose of water, after adding nitric acid of 1N so that pH may be set

to four to the obtained filter cake -- boehmite -- sol was prepared.

[0052] thus, the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.1%. boehmite -- it was 22cP when the viscosity of sol was measured. this boehmite -- the hydrated alumina produced by drying sol is boehmite.

The average pore diameters were 21.5 nm, and pore volume was 0.895 cc/g.

[0053] As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0054] Like this gestalt, there is a merit at the time of changing an addition order in the ability to raise the hydrated alumina slurry concentration obtained by neutralization. That is, according to this example, in the state of a hydrated alumina slurry, that whose aluminum₂O₃ conversion concentration was 1% can be raised even to 3% by working example 1-5 and the comparative examples 1 and 2. Although it is also possible to raise concentration by filtration concentration, as for a hydrated alumina slurry, it is preferred that water heat treatment carries out as it is fundamentally, and it can attain the miniaturization of a hydrothermal-reaction device by having raised the concentration of the hydrated alumina slurry in this gestalt.

[0055] [Working example 7] Although the amorphous hydrated alumina slurry was adjusted with the neutralization reaction with basic aluminum chloride solution and sodium aluminate solution and water heat treatment of it was carried out in working example 1, the example using the neutralized slurry to which the crystalline substance was intermingled in part in the hydrated alumina slurry is explained below.

[0056] First, the amorphous hydrated alumina slurry obtained by the method of working example 6 was neglected under the room temperature for 166 hours. When X diffraction measurement of

this neglected hydrated alumina slurry was performed, it was admitted in part that the crystalline substance (bayerite aluminum (OH)₃) was generating.

[0057]performing the same processing as working example 6 using this hydrated alumina slurry -- boehmite -- sol was obtained. pH of the boehmite slurry after 10.4 and water heat treatment of pH of the hydrated alumina slurry before water heat treatment was 12.2. the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.3%, and viscosity was 18cP. this boehmite -- the average pore diameters of the boehmite produced by drying sol were 31.0 nm, and pore volume was 0.695 cc/g.

[0058]As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0059]thus -- since bayerite will disappear and will serve as a boehmite single phase, if it processes on the hydrothermal conditions concerning this invention even if bayerite generates although the bayerite of a crystalline substance generates a part if the alumina slurry obtained by neutralization is neglected -- boehmite -- sol can be manufactured. In this case, there is a tendency for boehmite crystal growth to become early compared with the time of using an amorphous slurry.

[0060]On the other hand, when it separates from the hydrothermal conditions of this invention (i.e., when the case where pH falls before and behind hydrothermal, and hydrothermal temperature are less than 120 **), bayerite will remain, but such an example is later mentioned as the comparative example 3.

[0061] [Working example 8] In each of above-mentioned working example, although nitric acid was used as a dispersing agent, the example using acetic acid as a dispersing agent is explained below.

[0062]Neutralization reaction and hydrothermal reaction were

performed by the same processing as working example 7, and the boehmite cake was obtained. adding and carrying out amalgam decomposition of optimum dose of water, after adding acetic acid of 4N so that pH may be set to four in this cake -- boehmite -- sol is prepared. the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.1%, and viscosity was 16cP. this boehmite -- the average pore diameters of the boehmite produced by drying sol were 18.2 nm, and pore volume was 0.766 cc/g.

[0063]As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0064] [Working example 9] Next, the example using chloride as a dispersing agent is explained below.

[0065]performing the same processing as working example 8 except having changed into the chloride of 4N the acid used for amalgam-decomposition distribution from acetic acid of 4N -- boehmite -- sol is prepared.

[0066]the obtained boehmite -- the aluminum₂O₃ conversion concentration of sol was 20.1%, and viscosity was 18cP. this boehmite -- the average pore diameters of the boehmite produced by drying sol were 18.1 nm, and pore volume was 0.769 cc/g.

[0067]As a result of preparing coating liquid like working example 1, manufacturing a recording form and doing a printing test to this recording form, there is no blot and it was checked that ink absorbency is large.

[0068] [Comparative example 1] To 5000-ml glass beakers, aluminum₂O₃ conversion concentration =23.6%, Cl/aluminum mole ratio = until pH is set to 9.0, taking optimum dose of water for the basic aluminum chloride solution (trade name: made in [Daimei Telecom Engineering Chemical industry] the alpha yne 83) of 0.501, and stirring this solution by a homomixer, A sodium aluminate solution (aluminum₂O₃ conversion concentration = 1% and Na/aluminum mole-ratio =2.0) is gradually added under a room temperature, and an amorphous

hydrated alumina slurry is obtained. Next, hydrothermal reaction of 8 hours is performed for this slurry at 150 ** using autoclave. It was 4.8 when pH of the slurry after hydrothermal reaction was measured. Next, filtration washing of the slurry after hydrothermal reaction is carried out until the electrical conductivity of filtrate becomes below 20microS/cm. next -- adding and carrying out amalgam decomposition of optimum dose of water, after adding nitric acid of 1N so that pH may be set to four to the filter cake produced by doing in this way -- boehmite -- sol is prepared.

[0069] Thus, the aluminum₂O₃ conversion concentration of the obtained sol was 19.1%. It was 930cP when the viscosity of this sol was measured. Although the hydrated alumina produced by drying this sol was boehmite, those average pore diameters were 9.0 nm, pore volume was 0.444cc/g, and all were small values.

[0070] Thus, since it was the conditions that pH after a neutralization reaction was 9.0, and pH after hydrothermal reaction fell in the comparative example 1, After preparing coating liquid like working example 1 and manufacturing a recording form, as a result of doing a printing test to this recording form, it was checked that a blot arises and it is inferior to ink absorbency.

[0071] [Comparative example 2] Except having made water heat treatment by autoclave into 8 hours at 110 **, the same processing as working example 1 was performed, and alumina sol was prepared. Since the hydrated alumina produced by drying this sol has temperature conditions of hydrothermal reaction as low as 110 **, they are boehmite and a mixture of bayerite (aluminum (OH)₃).

Since the bayerite generated with boehmite was an about several micrometers big and rough particle, it was not able to prepare uniform sol.

[0072] [Comparative example 3] Using the slurry which was obtained in working example 7 and which the bayerite of the crystalline substance generated in part, except having carried

out water heat treatment at 110 °C for 17 hours, the same processing as working example 7 was performed, and alumina sol was prepared. The hydrated alumina produced by drying this sol is a mixture of bayerite and boehmite, and was not able to prepare uniform sol.

[0073] [Other working example] According to examination which the invention-in-this-application person other than above-mentioned working example performed repeatedly. A Cl/aluminum mole ratio For example, a neutralization reaction with the basic aluminum chloride solution from 0.45 to 1.5, and sodium aluminate solution, They are pH ten or more alkali fields about the amorphous hydrated alumina slurry preferably generated by neutralization reaction on conditions 40 °C or less, And if amalgam decomposition of this boehmite slurry is carried out after performing water heat treatment at the temperature of not less than 120 °C and obtaining a boehmite slurry, Viscosity when sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration is 100 or less cP, and boehmite -- the average pore size of the fine pores formed in the boehmite impalpable powder produced by drying sol -- not less than 15 nm -- pore volume -- the boehmite of 0.5 or more cc/g -- sol being manufactured and, When Information Storage Division by the ink jet method was performed, it has checked that the recording medium which can perform high record of grace could be manufactured.

[0074] Here, as a recording medium, what formed the ink absorbing layer in the paper as a substrate, the thing in which the ink absorbing layer was formed on the still more transparent plastic sheet for OHP as a substrate, etc. can be manufactured.

[0075] the boehmite used for manufacture of a recording medium -- it has checked that it was preferred to set viscosity when sol concentration is made into 20 % of the weight by aluminum₂O₃ conversion concentration to 50 or less cP as sol, and it was preferred that sol concentration is 18 % of the weight or more by aluminum₂O₃ conversion concentration.

[0076]

[Effect of the Invention]the boehmite used for manufacture of the recording medium for ink jets in this invention as explained above -- in manufacturing sol, They are pH ten or more alkali fields about a neutralization reaction with basic aluminum chloride solution and sodium aluminate solution, and the amorphous hydrated alumina slurry preferably prepared by neutralization reaction on conditions 40 ** or less, and -- carrying out amalgam decomposition of this boehmite slurry, after performing water heat treatment at the temperature of not less than 120 ** and obtaining a boehmite slurry -- boehmite -- in order to obtain sol, even if sol concentration is high -- boehmite with low viscosity -- sol can be manufactured. such boehmite -- when sol performs Information Storage Division by the ink jet method, it can manufacture efficiently the recording medium which can perform high record of grace.

[0077]Even if a part of hydrated alumina slurry prepared by neutralization reaction with basic aluminum chloride solution and sodium aluminate solution is crystallizing, if it is set as the conditions which become higher than pH of the hydrated alumina slurry before pH of the boehmite slurry after performing water heat treatment performs water heat treatment, even if sol concentration is high -- boehmite with low viscosity -- sol can be manufactured. such boehmite -- when sol also performs Information Storage Division by the ink jet method, it can manufacture efficiently the recording medium which can perform high record of grace.

[Translation done.]